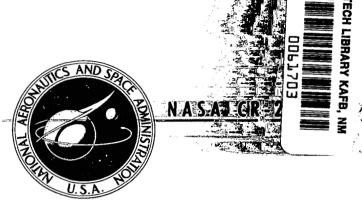
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SITE ALTERATION EFFECTS
FROM ROCKET EXHAUST IMPINGEMENT
DURING A SIMULATED VIKING MARS LANDING

Part II - Chemical and Biological Site Alteration

R. R. Husted, I. D. Smith, and P. V. Fennessey

Prepared by

MARTIN MARIETTA CORPORATION

Denver, Colo. 80201

for Langley Research Center

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION . WASHINGTON, D. C. . MARCH 1977



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1. Report No.	2. Government Access	ion No.	3. Recipie 0061703
NASA CR-2814			
4. Title and Subtitle SITE ALT	ERATION EFFECTS	FROM ROCKET	5, Report Date
EXHAUST IMPINGEMENT			March 1977
MARS LANDING - Part Site Alteration	II - Chemical a	nd Biological	6. Performing Organization Code
7. Author(s)			8. Performing Organization Report No.
R. R. Husted, I. D.	Smith and P. V.	Fennessey	10. Work Unit No.
9. Performing Organization Name and Ad	dress		
Martin Marietta Cor	poration	Ì	11, Contract or Grant No.
Denver Division			NAS1-9000
P. O. Box 179 Denver, Colorado 80	201	ł	13. Type of Report and Period Covered
12. Sponsoring Agency Name and Addres			
National Aeronautic	s & Space Admini	stration	Contractor Report
Langley Research Ce			14. Sponsoring Agency Code
Hampton, Virginia 2	3665		l
15. Supplementary Notes			
Langley Technical Monit	or: Arlen F. Carte	r	}
Final Report			
16. Abstract			
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17. Key Words (Suggested by Author(s))	chemical	18. Distribution Statemen	nt
alteration	biological	Unclassifie	d - Unlimited
rocket	hydrazine		
Mars	fue1		
landing site	purified		Subject Category 28
SOIL	contamination	21 No of	
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^{*} For sale by the National Technical Information Service, Springfield, Virginia 22161

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SITE ALTERATION EFFECTS FROM ROCKET EXHAUST IMPINGEMENT DURING A SIMULATED VIKING MARS LANDING

Part II. Chemical and Biological Site Alteration

by R. R. Husted, I. D. Smith and P. V. Hennessey

SUMMARY

A potential interference problem for the Viking '75 scientific investigation of the Martian surface resulting from retrorocket exhaust plume impingement on the surface was investigated experimentally and analytically. Tests with a full-scale terminal descent engine under simulated Martian atmospheres, pressures and soils were conducted at the NASA/Johnson Space Center, White Sands Test Facility to determine the extent of chemical and biological site alteration. These tests were conducted with a descent engine redesigned from a conventional bell nozzle to a multiple nozzle configuration consisting of 18 small bell nozzles. An earlier test program to define the physical site alteration led to the engine redesign. This effort is described in reference 1.

The chemical test involved direct measurement of the effects of the interaction of the landing rocket plume gas field with a nominal soil model. The results indicate that an engine burning standard Military Specification hydrazine fuel will contaminate the landing site with ammonia (50-500 ppm), nitrogen (5-50 ppm), aniline (0.01-0.5 ppm), hydrogen cyanide (0.01-0.5 ppm), and water (quantity not measured). A purified fuel, with impurities (mostly aniline) reduced by a factor of 50-100, limits the amount of hydrogen cyanide and aniline to below detectable limits and leaves the amounts of ammonia, nitrogen and water in the soil essentially unchanged. Large amounts of ammonia trapped in the soil will make interpretation of the organic analysis investigation more difficult; however, with no obvious alternate systems, the Molecular Analysis team has accepted the ammonia contamination for Viking. However, the team is conducting experiments to fully understand the effects of ammonia in the soil on organic analyses. Nitrogen and water concentrations in the soil are questionable because of the experimental difficulties in measuring these species.

The biological tests indicate that the combined effects of the plume gases, surface heating, surface erosion and the gas composition resulting from the retrorockets do not interfere with the Viking biology investigation. This was shown by Biology team laboratory testing to determine the soil microbial population before and after engine firing.

INTRODUCTION

A primary mission objective of Project Viking is the search for extraterrestrial life through a scientific investigation of the Martian surface (see reference 2). The Viking Lander, shown in figure 1, incorporates propulsive terminal descent in order to soft-land the scientific payload. The general question of native surface modification by the engine plumes had to be examined closely in order to establish the relevance of the returned scientific data. It was found that the descent engines had to be modified to achieve an acceptable level of landing site alteration.

A multiphase test program has been conducted to investigate the amounts of physical, chemical, and biological alteration of simulated Martian surfaces that would occur as a result of the terminal engine plumes and to investigate vehicle design changes that could be made to minimize these site alterations. The baseline design of the Viking lander called for the use of three monopropellant engines with conventional bell nozzles having an area ratio of 20 to 1. An impingement pressure test of a full-scale prototype terminal descent engine in a simulated Martian environment was conducted at the NASA/Johnson Space Center, White Sands Test Facility (WSTF) during the Phase IA tests to determine the radial variation of surface impingement pressure as a function of engine altitude above the surface (reference 3). Soil erosion tests were then conducted in the NASA/Langley Research Center 60-foot vacuum sphere using sub-scale and full-scale cold-gas jets, modeled to match hotjet pressures, to determine the severity of the physical alteration that could be expected. These tests indicated that physical erosion of simulated Martian soil by the exhaust plume of an engine with a conventional nozzle descending toward the surface would not be acceptable.

A comprehensive Phase II Site Alteration Test Plan was then formulated to evaluate candidate engine/nozzle performance against established criteria (reference 4). When it became apparent that engines with conventional bell nozzles would not meet the established physical criteria, sub-scale cold-gas nozzle tests were conducted at the NASA/Langley Research Center (LRC) and at the Martin Marietta Corporation (MMC) to develop nozzle designs which would reduce surface impingement pressures. The LRC tests were conducted in the 60-foot vacuum sphere under simulated Martian pressure conditions. The MMC tests were conducted in the Denver Division Cold Flow Laboratory under earth ambient conditions. Based on the results obtained from these sub-scale cold-gas tests, full-scale engine nozzle configurations were designed and fabricated. These nozzles were tested on a "boilerplate" engine which could accommodate nozzle changes. Flat plate impingement pressure tests together with soil impingement tests were conducted under simulated Martian pressure and carbon dioxide environments in a test chamber at the NASA/Johnson Space Center, White Sands Test Facility. Flat plate impingement pressures and temperatures of the engine exhaust were evaluated for 10 different nozzle configurations at a lander

descent velocity of 1.52 m/sec. For promising engine nozzle configurations, impingement effects of the engine exhaust on simulated Martian soils were evaluated to determine soil cratering, soil transport and deposition, temperature profiles, and soil chemical and biological effects. From an evaluation of these test data a multiple nozzle configuration, consisting of 18 small bell nozzles, was selected for use on the Viking Lander.

Once the design of candidate engines had been established additional studies were necessary to determine the extent of the Mars landing chemical and biological site alteration using the full-scale prototype engine mounted on a 1/3-segment of the Viking lander. These full-scale tests were part of Phase II conducted under simulated Mars pressure and carbon dioxide environments at the WSTF 302 test chamber (reference 5). The Biology and Molecular Analysis science teams helped formulate success criteria by establishing allowable limits of physical, chemical, and biological alteration.

SYMBOLS

OC Degrees centigrade

cm Centimeters

GC Gas chromatogram

HCN Hydrogen cyanide

H₂ Hydrogen

kg Kilogram

m Meters

mb Millibar

m/e Mass to electronic charge ratio

mg Milligrams

Mil-Spec Military Specification

ml Milliliter

mm Millimeters

m/sec Meters per second

N Normal with units of equivalent weights per liter

NH₃ Ammonia

N₂ Nitrogen

N₂H₄ Hydrazine

ppb Parts per billion

ppm Parts per million

ppm/hr Parts per million per hour

psig Pounds per square inch, gauge

R Right side of biology tray

TSA Trypticase soy agar

UV Ultraviolet

 μg Micrograms

μ1 Microliters

< Less than

TEST APPARATUS AND PROCEDURES

White Sands Test Facility - Phase II Tests

For the WSTF Phase II site alteration tests a 6.1-m section was added to the cylindrical part of Test Stand 302 vacuum chamber to extend the usable height to 14.3 m. The additional height was required in order to simulate the significant part of the exhaust/soil interaction which occurs during the terminal descent phase of the Viking landing. For these tests, the use of 3 full-scale rocket engines was impractical because of facility volume limitations; therefore, a 1/3-segment of the Viking Lander was simulated with one full-scale engine attached and, as shown in figure 2, reflection planes 4.9-m high were used to simulate the jet interaction effects of the other two engines. The 1/3 lander segment was attached to a carriage designed to ride smoothly on a vertical track. Travel of the carriage, including starting, the constant velocity descent, and stopping, was controlled by a hydraulic motor and an electro-mechanical brake through a chain and sprocket drive mechanism. A potentiometer attached to the upper sprocket of the chain drive assembly was used to record lander position on the track as a function of time. From these data the lander descent velocity was computed for each test.

Propulsion system - Two hydrazine fueled catalytic rocket engines were used for the Phase II tests. Initial exhaust gas sampling tests were conducted using a flight weight engine with a 20:1 area ratio conventional bell nozzle having a 15.2 cm exit diameter. The remainder of the sampling tests and the pressure and soil tests (see Table 1) were conducted using a "boilerplate" engine. This engine, which can be seen in figure 2, had a heavy wide-flange joint between the nozzle and the catalyst bed that made it possible to change nozzle configurations. Both engines were capable of producing 2669 newtons (600 lbs) of thrust, but were throttled to 667 newtons (150 lbs) during the Phase II testing to approximate the value of Viking Lander retrorocket thrust near the Martian surface. Engine performance was monitored by recording propellant tank pressure, engine injector manifold pressure, engine chamber pressure, and gas temperature in the engine below the catalyst bed.

<u>Propellant</u> - Using a simple hydrazine fuel (N₂H₄) where there is no organic carbon avoids the lunar sample contamination problem of Apollo LM where dimethyl hydrazine and dinitrogen tetroxide retrorocket fuel and oxidizer yielded compounds containing six or more carbon atoms (reference 6). Hydrazine is known to decompose in the presence of a catalyst according to the following equations:

The first reaction goes to completion whereas the amount of decomposition of ammonia (NH3) shown in the second reaction is highly dependent upon the engine design and use.

A series of studies involving the interaction of plume gases with the Martian carbon dioxide atmosphere (references 3 and 7) showed a mole percent composition in the plume of approximately 30% nitrogen, 30% ammonia, and 40% hydrogen. A measurable amount (0.2%) of hydrogen cyanide (HCN) was also detected in the plume. Investigation into the purity of standard hydrazine produced the following chemical analysis:

Species	Specified Concentration (%)	Observed Average Value (%)
Hydrazine	97.5	98.0
Water	<2.5	1.5
Other	-	0.5

Discussions with a hydrazine manufacturer led to the identity of the "other" species as aniline (C_6H_7N) with a possible trace of heptane (C_7H_{16}). These chemicals are added to the fuel during the manufacturing process. The possibility that hydrogen cyanide is directly related to the aniline impurity led to the purification of hydrazine fuel (45-90~kg) by fractional crystallization. The stages of purification were followed by measurement of the concentration of aniline using its UV absorption spectrum, which is adequate to the 1-10 ppm level. To ensure that the process removed all organic impurities and did not add any new compounds an independent check was accomplished using a trace carbon-hydrogen analysis. The results of the total carbon tests and the aniline test done at manufacture and before the test are:

Hydrazine Fuel Impurities (ppm) .

Туре	Total Carbon	Aniline at Manufacture	Aniline Before Test Firing				
Mil_Spec	8000	4200	4000				
Purified (Average)	73	35	78				

These results indicate that although aniline is not the only impurity in the fuel the purification process removes all the carbon containing compounds in approximately the same proportion.

Both Mil-Spec and purified hydrazine as described above were used for the Phase II tests in order to quantify their effects on the Viking molecular analysis and biology experiments.

Soil containers -

Soil bed: The main soil bed used for physical site alteration measurements is described in reference 1.

Soil trays: Along one side of the soil bed were two soil trays which extended along radial lines from engine centerline. One tray which adjoins the reflection plane extended 4.6-m beyond the soil bed container for a total distance from the engine centerline of 5.9-m. The second tray extended 4.4-m beyond the soil bed for a total distance of 6.8-m from the engine centerline. These trays contained sealable soil cups for measuring chemical contamination of the soil, open soil cups for measuring soil erosion and transport, and unobstructed areas for assessing the effect of engine plume gases on prepared surface morphological features.

A third soil tray adjoining the engine reflection planes on the opposite side was used solely to assess biological degradation as a result of engine plume impingement. All three side trays were 7.6-cm deep.

The distribution of three adjoining soil trays together with the main soil bed constitutes most of the sampling area available to the Viking surface sampler. This geometry was used so that the symmetry of plume/soil interaction could be assessed throughout the sampling area to determine if certain areas were more

favorable for sampling. The schematic of figure 3 illustrates the main soil container, soil trays, and the area available to the surface sampler.

Open soil cups: Open soil cups were placed in the soil trays to permit measurements of soil transport and deposition during severe soil disturbances. After the first engine firing in soil, test 12A, the open soil cups were discontinued because the soil erosion and transport were primarily due to viscous shear and the very small amount of collected material was assessed to be insignificant in value.

Sealable soil cups: Two types of remotely controlled sealable soil cups were used in the Phase II tests. One type, shown in figure 4, was filled with soil prior to testing and sealed during the test either just before or just after engine firing. The other type was used in conjunction with an auger which dug and delivered a soil sample from the main soil bed shortly after engine firing.

The prefilled sealable soil cups were 10.2-cm in diameter and either 5.1, 10.2, or 15.2-cm in depth. The upper part of the cups and the cap assemblies were the same for each size. An 80-mesh stainless steel screen 5.1-cm in height with a removable screen top formed the upper part of the cups. The cups were sealed into the caps with a neoprene "0" ring which was covered with a teflon ring that was displaced during sealing. This teflon ring was required to prevent soil contamination of the sealing surface. The "0" ring was sprayed with a dry fluorocarbon before installation to facilitate sealing. The caps and cups were equipped with bellows seal valves so that a gas phase analysis could be conducted.

Two types of actuators were used for closure and sealing of the cups. One type, shown in figure 5, holds 3 cups and was used in the soil trays to seal cups immediately after engine firing. The cups were held in the sealed position until a post-test crew could secure the seal. The second type of actuator shown in figure 6 was principally used to seal control sample cups, which were open to the test chamber environment. Immediately before engine firing the cups were sealed and snap latches mechanically secured the seal until the set screws were tightened by a post-test crew.

The auger-mechanism sealable cup was approximately 6.3 cm in diameter with an inside depth (usable soil space) of 14 cm. It was also fitted with a bellows seal valve to permit in situ gas phase analysis of the contents. A small neoprene "0" ring coated with a dry fluorocarbon was used to provide a good seal. At the open end of the auger cup, a two-faced bit was attached by screws to the cup as a soil gathering device (see figure 7). The performance of the auger system was less than satisfactory.

All sealable cups were disassembled and cleaned to the requirements of JSC Specification MSC-00066 level CP-7. This specification (as developed for lunar tools and lunar sample containers) consisted of pre-clean with acid followed by ultrasonic cleaning in filtered freon. Cleaned cups were bagged in teflon and then double-bagged in polyethylene.

After cleaning, the cups were reassembled in a class 100,000 clean room and loaded with soil. Throughout all handling, complete clean room clothing (hood, coveralls, overshoes) and teflon gloves were used.

Before soil loading each cup was vacuum-leak checked. An increased leak rate was associated with repeated cleanings caused by the severity of cleaning on parts whose tight fit was necessary for a good seal, including valve bellows and stems.

Soil was weighed and packed into the cups to achieve a density of approximately 1.7 g/cm³. Cups and caps were bagged separately in teflon and delivered to the test chamber where they were installed using teflon gloves. New neoprene ''0'' rings and Kel-F valve seals were used on each run.

Auger cups were cleaned and leak-checked in the same manner. They were assembled in the clean room and bagged in teflon until installation.

Soil models - The soil types used in the Phase II chemical and biological site alteration tests are based on the specifications in reference 8. Lunar nominal is based on the only real data from an extraterrestrial body and one, where the dominant surface morphology like Mars is characterized by impact craters. This soil model was well-graded and contains a particle size distribution from submicron size particles to a very coarse fraction. The rationale for using the lunar nominal soil model in the Phase II tests is discussed in reference 9.

The other soil model was a natural occurring volcanic sand dune model which, with respect to chemical and biological site alteration, was used only for ammonia absorption analysis. It was used for this purpose because of its high porosity and permeability. The origin of the sand dune material is pyroclastic debris ejected from Sunset Crater around 600 A.D. This coarse mafic scoracious debris has been well-sorted by wind action and has a mean particle size of about 0.5 mm.

Test preparation and procedures - Test conditions for the biological and/or chemical tests conducted for Phase II at the WSTF 302 vacuum chamber are presented in Table 1. Test numbers refer to the type of test described in reference 10 and not to the order in which they are conducted. For the biological and/or chemical tests the program can be divided into 2 types of tests conducted under simulated Martian conditions.

- a. Static firing of engine to collect exhaust gas samples for analysis.
- b. Engine descending with plume impinging on a bed of soil.

Exhaust gas sampling tests: For the majority of the tests the engine was fixed either 1.5 or 3.0 m above the blast plate depending on the engine nozzle configuration. Gas samples were taken for 12 seconds starting at 2 and stopping at 14 seconds after engine firing.

For tests 11J and 29, the engine was operated while descending at approximately 1.5 m above the blast plate and ended at engine cut-off. Sampling duration was 5 seconds during which time the nozzle descended to 0.76 m above the blast plate, stopped descending, and then engine cut-off occurred approximately 4 seconds later.

The gas sampling equipment shown in figures 8 and 9 was used for the majority of the tests. Just prior to chamber door closure, the manual valves on the sampler were opened and the blast plate was cleaned with lunar tool quality trichlorotrifluorethane. The test chamber was evacuated to 0.16 Torr and backfilled with laboratory grade carbon dioxide to the pressure levels shown in Table 1. During chamber evacuation, liquid nitrogen flowed into the cold trap. Cold trap temperature was monitored by a thermocouple to verify that it was at -196 + 14°C for a minimum of 30 minutes prior to engine firing. Following engine firing, both the test chamber and the gas sampler were backfilled with gaseous nitrogen to ambient pressure in order to minimize any pressure differential between the two, thus, minimizing gas exchange between the sampler and chamber through system leaks. Solenoid valve closure resulted in gas sampler pressures ranging from ambient pressure to 52 Torr less than ambient pressure. The backup manual gas sampler valves were closed as soon as safety would permit following opening of the test chamber doors. The contents of the traps were analyzed in the laboratory for hydrogen cyanide and ammonia by using gas chromatography.

The hydrogen cyanide analysis was performed using a Franklin and Marshall gas chromatograph equipped with a 2.4 m, 0.16 cm diameter PAR-1 column and flame ionization detector. A 5-ml sample at 20 psig (nominal) was used to perform the analysis. The following column temperature program was used:

- a. 0 °C (wet ice) for five minutes.
- b. 44 °C for one minute after a two minute stabilization period.
- c. Program temperature at 8 °C per minute to 212 °C.

The ammonia analysis was performed using a PE-800 gas chromatograph equipped with 3.0 m, 0.32 cm diameter Chromosorb 103 column and a thermal conductivity detector. A 10-ml sample at 30 psig was used. The analysis was performed under isothermal conditions at $150\,^{\rm O}{\rm C}$.

The gas sampling equipment shown in figures 10 and 11 was used for tests 11J and 29. The procedure was similar to that described for the previous exhaust gas sampling tests.

Chemical alteration of soil: The test chamber and its associated facilities at White Sands were not organically clean. Since it was impractical to clean the complete chamber and control the access into the chamber through-out all pretest operation, the soil cups and their associated apparatus were brought under a rigid control system. The cups were cleaned, stored, filled, and used in a standard manner in each test and between tests.

The soil cups were placed in the trays the evening before or the day of a test run and a need-only access was then imposed on people entering the chamber. This was to minimize the chance of accidental contamination. Control cups were used and were closed just before engine firing. As soon after firing as possible, all other cups were sealed to the ambient environment in the chamber. All cups were removed from the chamber on the day of the test and taken to the chemistry laboratory for analysis.

Biology alteration of soil: The main emphasis of the site alteration biology test was to assess the effect of the Viking lander rocket exhaust products on the microbe population in lunar nominal soil under simulated Martian atmospheric conditions and to demonstrate by analysis and experiment how the site alteration biology test results compare to the accepted criteria presented in reference 4.

The initial phase of the biology test demonstrated the presence of sufficient and predictable numbers of organisms in the test soil prior to the engine firing. This was accomplished by sampling and analyzing containers of lunar nominal by plate counting techniques for uniformity in numbers of microbes/mg of soil.

After establishing the baseline microorganism population, the soil was used to prepare the biology tray in chamber 302 (figure 3). The biology tray was approximately $0.61 \times 2.4 \times 0.076$ m deep with its longest dimension parallel to a radius from the centerline of the engine as shown in figure 3. During preparation of the biology tray, the traffic in the test chamber was minimized. The tray was wiped with isopropyl alcohol as were the tamping tools, shovel and tray cover. All personnel preparing the tray wore sterile gloves. The surface of the soil in the tray was at the same height as that of the other soil trays.

Pre- and post-test sampling from the biology tray was accomplished by an individual wearing a pair of sterile gloves and using a sterile tongue depressor to push the surface soil into the sterile sampling tubes. A fresh sterile tongue depressor was used at each change in distance from the centerline of the engine. Core samples were taken by inserting the tube vertically into the soil 3.8 to 5.1 cm using a twisting action. The tube was removed using a scooping action to minimize loss of sample and the surface was leveled using the tongue depressor employed for surface sampling. Also surface samples were taken for the four Viking biology experiments (Gas Exchange, Labeled Release, Light Scattering and Pyrolytic Release) just prior to closing the chamber door and again as soon as safety would permit after each firing. Just the top few millimeters of soil were collected.

The appropriate samples were transferred to the four Viking biology experimenters for evaluation with their experiments. The remaining samples were assayed by the plate count technique to determine their microbial burden.

Each half-gram soil sample was placed in 10 ml of thioglycollate broth, mixed for 10 seconds with a Vortex mixer and ten-fold serial dilutions were made from this initial tube; one ml into nine ml of broth. Trypticase soy broth was used for diluting soil to be cultured aerobically and thioglycollate broth was used for anaerobic dilution tubes. The soil dilutions were plated aerobically using 0.1 ml samples on the surface of trypticase soy agar plates. The plates were inoculated in triplicate and the 0.1 ml of fluid was spread on the surface of each plate using a sterile glass "hockey-stick". Anaerobic pour plates were made in triplicate of each dilution using one ml sample/plate and molten thioglycollate agar (3% thioglycollate broth, 2% agar). The anaerobic plates were placed in Brewer anaerobic jars as soon as they solidified and were incubated in an anaerobic hydrogen and carbon dioxide environment. All plates were incubated at 25 °C for 72 hours.

After counting the plates the counts and sample weights were entered in a calculator programmed to compare the means of the control and test group. The comparison was made according to the t-test for the null hypothesis at the 95% confidence level on the basis of a two-sided alternative (reference 11). The null hypothesis (equal control and test sample means) was rejected only when the tabular value for t was exceeded by the absolute value for t. Where the value for t was too high the mean for the control sample was reduced by 50% (see biology criterion, reference 4) and the control sample variance was divided by 4 and t was recalculated. The biology criterion was met in every case.

RESULTS AND DISCUSSION

Exhaust Gas Sampling Tests

A series of gas phase samples were taken during the site alteration tests in an attempt to isolate the source of the cyanide. The first of these was devised to establish the difference between purified and Mil-Spec fuel, and to learn the effect, if any, on the cyanide concentration in the plume of engines having different nozzle configurations. The results of these tests presented in the following table in light of the analysis of the impurities in Mil-Spec and purified hydrazine previously presented show that there is a direct relationship between the carbon content of the fuel and the amount of hydrogen cyanide formed. The data also revealed that neither the amount of plume mixing with the ambient atmosphere nor the actual nozzle configuration affected the hydrogen cyanide concentration.

Gas Phase Test Results

Test No.	Fuel	Nozzle	Pressure (mb)	HCN (mg/2.8 liter)				
9	Mil-Spec	20:1	4.5	21.9				
10	Mil-Spec	20:1	10.0	31.7				
2	Pure	20:1	3 . 3	0*				
3	Pure	20:1	9. 9	0				
3C	Pure	20:1	4.9	0				
3B	Pure	7	2.8	0				
3A	Pure	24	8.9	0				

^{*}Detection limit 0.1 mg.

During the course of the soil analyses some question was raised as to the value of the reported exhaust gas trap measurements. The ammonia and hydrogen cyanide removal efficiency was checked in the following manner. The gas collection system used above and a ten-liter "watermelon" were evacuated (10^{-6} Torr) and backfilled with 325 μ g of hydrogen cyanide and 17.0 mg of ammonia (the quantities injected were also verified by independent gas chromatographic analysis). The vessels were then pressurized to 10 Torr with carbon dioxide and allowed to stabilize overnight. The containers were analyzed by sweeping the contents into a water trap using helium gas flow and finally by washing the traps with a water solution. The amounts of hydrogen cyanide and ammonia were determined and a recovery factor of approximately 85% was found for both gases.

These same traps were used during the Mil-Spec and the purified fuel test run and the concentrations of hydrogen cyanide determined are listed below. The data verified the initial results.

Test No.	Fuel	Nozzle	Pressure (mb)	HCN (mg)		
12G	Mil-Spec	Fluted	11,5	0.16		
29	Pure	18	9. 5	0.0*		

^{*}Detection limit 0.005 mg.

The data acquired during these tests support the theory that the hydrogen cyanide does come from the carbon impurities in the fuel. The results of these tests led to the conclusion that the amount of hydrogen cyanide and aniline can be controlled and even eliminated by using purified fuel. One additional and more general point is that whenever a soft planetary landing mission is being planned a considerable amount of time and attention must be given to the possible interactions of the retrorocket exhaust gases with the atmosphere and surface, thereby possibly compromising the scientific investigations.

Chemical Alteration

The details of the interaction of the rocket plume gases and a soil model were of major importance in the Phase II site alteration program. Initial studies (references 3 and 7) indicated that the soil would be exposed to a number of gases including ammonia, nitrogen, hydrogen, water and hydrogen

cyanide. The question of how much of each gas would be trapped by a soil model and how each could affect the analysis of the soil was investigated. This was done using individual chemical tests for ammonia, hydrogen cyanide, aniline, and other organics. A numerical analysis was initiated to show the expected amount of hydrazine at the landing site.

<u>Soil models</u> - Lunar nominal was judged to best represent our limited knowledge of Mars. Enough testing was conducted with this soil to formulate some general conclusions concerning the interactions of the plume gas with improvement of each of the analyses. Because the majority of tests were carried out on just one soil model, there is some question as to how good an extrapolation can be made to the Martian case; but it does serve as a basis for any future work.

Random samples of the lunar nominal material were chemically analyzed by a soil analysis laboratory. The results are shown in Table 2 and indicate that the model is very adequate for ammonia, hydrogen cyanide and hydrazine studies. However, the relatively high organic carbon content (240-288 ppm) indicated that contaminating organic compounds can be found only by looking at differences in the control and test samples. One further effect of the high level of organics is that the absolute amount of contamination added can never be obtained from these tests. An additional test was carried out to define the extent of the organic interference.

A sample of the lunar nominal material (1-10 mg) was placed in the probe of a mass spectrometer and inserted directly into the source and heated. A spectrum was taken every ten seconds from the time of insertion until the sample temperature reached 250 °C. These spectra were then analyzed by computer. The results showed that there were no major compounds (other than air and water) in the soil. At the highest temperature (250 °C) some very small peaks (<1%) at m/e 97 and 98 could be detected. These tests showed that the lunar nominal soil should be adequate for the site alteration tests.

<u>Soil ammonia</u> - Test samples and controls were obtained using the hardware and soil preparation methods described earlier. The following paragraphs will cover the methods of analysis, the data from each test and the conclusions drawn from the total information.

It was shown in the gas phase tests that the plume gases contained approximately 30 mole per cent ammonia; therefore, in the selection of soil ammonia analysis methods, the emphasis was on reproducibility and convenience and not on sensitivity. The two methods selected for parallel development were a standard Kjeldahl and a Nesslers. This development included the following steps for each reagent:

- a. Develop a set of test standard and calibration curves.
- b. Run interference tests with hydrogen cyanide.
- c. Use the methods on the untreated soil model (control).
- d. Analyze soil treated with known amounts of ammonia.

The results of this program showed that the Kjeldahl method was accurate and sensitive from a lower limit of 1-10 ppm to very high concentrations (30-50% by weight). The Nesslers method was very accurate in the lower ranges of concentration (high ppb to 1%). Since both methods were adequate in the expected soil concentration ranges, the choice to use the Kjeldahl analysis as the primary source and the Nesslers as a back-up was based on consideration of convenience and laboratory experience of the analyst at the White Sands Laboratory. The interference tests showed that the hydrogen cyanide did affect the ammonia titration, but boiling with 50% by weight sodium hydroxide solution reduced this effect to a non-interfering level. The soil blanks showed an average background of 1-3 ppm ammonia and the tests run on soil treated with known amounts of ammonium chloride showed a 50% or better recovery.

The general procedure used in handling the soil during an analysis is listed below;

- a. The cups were purged with helium and the gas bubbled through 0.1N sodium hydroxide solution and vented to the atmosphere.
- b. The cups were opened and the soil transferred to a 2-liter bottle and covered with a sodium hydroxide solution (from first step plus enough additional solution to cover the soil to a depth of 5 cm).
- c. The solution-soil slurry from the second step was agitated by bubbling helium through it for 24 to 48 hours.
- d. Clear solution above the soil layer was drawn off for analysis.

A total of six tests were run where ammonia concentrations were measured from soil placed in the soil cups or in the main soil bed. The data for these runs are tabulated in Table 3 and show two different levels of ammonia in the soil. The first range of concentrations is obtained from the soil samples which were exposed to the plume gases for less than thirty seconds. This level (\sim 125 ppm) would represent a minimum amount of ammonia that could reach the soil and can be assumed to be a result of direct plume impingement in the sampling area. This

type of condition would be expected from a landing in a wind which could quickly dissipate the plume gases. The second or upper limit was obtained from soil in cups which remained open for more than 10 minutes. The large increase in concentration to the 300-500 ppm levels represents a combination of soil sorption from both a direct plume flow encounter and a high concentration of ammonia in the atmosphere after engine cutoff. Figure 12 shows a graphic representation of these two clusters of data with a curve drawn through the higher concentration set.

An additional refinement of the data was made during the analysis of selected soil cups. This was accomplished by dividing the soil into layers in the approximate ratio of 1:2:3:4 by weight and analyzing each layer for its ammonia concentration. For example, in Test 12D a sample taken 3.0 m from the engine centerline shows an average concentration of 56 ppm ammonia. The individual analyses of the soil layers shows a concentration in the layers of 210:21:33:48 ppm, respectively. This indicates that the ammonia is being held in the upper 1 cm of the soil. A curve showing a plot of all data gathered in this fashion is given in figure 13.

There are two important conclusions that can be drawn from the data presented in figures 12 and 13. The first is that the ammonia concentration in the soil will probably be in excess of 100 ppm for a large distance (30 m) in all directions from the engine centerline. This is a result of the radial flow of the plume gas along the surface. In order to obtain a sample of soil with little or no increase in ammonia, it would have to be obtained from outside the plume flow field (60-150 m from engine centerline). The second conclusion is that the profile (with depth) of the ammonia in the soil is directly related to the porosity of the soil. Because of the fine particulate nature of the soil used in these tests most of the plume gases were trapped in the top few millimeters. Soils with higher permeability will correspondingly allow the gas to penetrate to a much greater depth. This trend was observed in the soil pore pressure results for the lunar nominal and dune sand soil models (reference 1).

Soil cyanide - The development of a method for the analysis of cyanide followed generally the same steps as for ammonia. A modification of the Epstein cyanide determination (references 12 and 13) was selected as the primary method and a silver nitrate titration was selected as a back-up. The calibration data showed that the analyses were sensitive to concentrations of HCN in the ppb range in standard solutions. When soil was introduced into the medium the sensitivity was reduced by some of the impurities leached out into the medium. The interference tests showed that ammonia in the solution reduced the sensitivity even more. In order to avoid this ammonia interference a step was added in the analysis in which the sodium hydroxide solution was boiled for 10 minutes. This reduced the ammonia concentration in the solution and left only a small soil interference.

Because of the low concentrations of cyanide found during the course of the tests, a number of checks and additional studies were carried out on each step of the analysis. Initial work showed that when a cyanide salt was added to the soil the recovery was better than 50%. In a later set of tests hydrogen cyanide gas was introduced directly into the soil and when this soil was analyzed a recovery of 15-25% was determined. This change might be attributed to a difference in the interaction between the gas and soil and between a salt and the soil.

Consideration was also given to the method of getting the cyanide out of the soil and into solution. As outlined in the discussion on soil ammonia, the soil was allowed to stand 24 to 48 hours with mild agitation. An alternate method was proposed in which a measured quantity of soil was boiled in a sulfuric acid solution. The comparison tests were run using blank soils, soils equally doped with cyanide and no soil. The results verified that the sodium hydroxide leach approach did free 15-25% of the cyanide in the soil with a basic background level of approximately $5-6\times10^{-8}$ gm of cyanide/gm of soil. The direct removal by boiling with sulfuric acid proved to be a complete failure, because of both an increase in basic background by a factor of three and by a 0% recovery factor.

A total of six engine firing tests were run where measurements of cyanide concentration were obtained. These data are tabulated in Table 4. The values given represent the actual laboratory finding; and, based on recovery tests, must be multiplied by a factor of 5 or 6 to obtain true values of cyanide in the soil. The general trend of increase seen in this table in going from early to later tests indicates the improvements made on the analysis procedures with time as well as nozzle and test condition differences. These data need to be interpreted with caution because of the large losses due to soil interference and the fact that the test values are very close to the controls. Even with this rather cautious approach to the interpretation there is a trend in the data leading to an apparent increase in the cyanide in the Mil-Spec fuel tests (12D through 12G). These data alone could not be considered strong enough evidence to cause a change in the choice of fuel; however, as shown in the next section the other fuel impurities do provide the strong evidence required for a change in the selected fuel.

The data gathered in test 12G (dune sand) is plotted in figure 14. The curve could be off by a factor of 2 or 3 but it does show the same general trend as did the ammonia, namely a higher concentration the closer to the engine centerline. More detailed tests would have to be performed before a clear understanding of cyanide variation could be obtained.

Soil aniline - Aniline, an organic compound with composition C_6H_7N , is the second largest impurity (water is the largest) in Mil-Spec hydrazine fuel. Its concentration range has been reported to be between 0.3 to 0.7% by weight. During the site alteration gas phase tests it was shown that some of the aniline does pass through

the engine catalyst bed (at 1000°C) and can be trapped and identified in the plume gas. For example, the two gas trap tests run with purified fuel showed a level of 10⁻⁶ppm aniline in the traps while the identical Mil-Spec fuel runs showed values for aniline ranging in the 10⁻⁴ppm level. The two order of magnitude change is in agreement with the basic aniline concentration change from purified to Mil-Spec fuel (50 ppm to 5000 ppm). Based on these data the presence of aniline in soil around the landing site should be measurable if Mil-Spec fuel is used.

There was some question whether or not aniline could even be seen in a soil because of its low concentration in the fuel. The initial set of tests was designed to look at a soil both before and after an engine burning Mil-Spec fuel was fired on it. The analysis involved the following steps:

- a. 10 gm of soil was extracted with 20 ml of water.
- b. The total water sample was then extracted with 20 ml of freon.
- c. The freon was evaporated to 200 μ l and a 40- μ l portion of this was injected into the gas chromatograph.

A series of calibration tests were made to show that the peak in the gas chromatogram identified as aniline was truly representative of aniline in the soil and not an artifact of the procedures or solvents used. The results of this work are shown in figures 15 through 19.

The basic retention time for aniline on the gas chromatograph was established by adding known amounts of aniline directly to the freon solvent and to a blank soil sample before extraction with water. The aniline peak appears in the time frame between 9.6 to 10.2 minutes (abscissa on figures 15 through 19) with a maximum at approximately 9.9 minutes. Enough of these calibration tests (figure 15) were made to establish a curve of peak area vs concentration in the freon. This was used in the interpretation of the later spectra. The chromatogram shown in figure 16 establishes a recovery value of 40-60% of aniline from the soil. This means that the concentration reported for the test soil is probably within a factor of three (on the low side) of the true concentration.

The chromatogram in figure 17 shows the aniline content of a soil taken directly under the engine centerline after a firing using purified fuel. The area between 9.6 and 10.2 minutes is virtually free of any peak; therefore, the soil contained an undetectable quantity of aniline (less than 0.02 ppm). The control from the purified fuel test also showed the absence of any aniline.

These results can be contrasted to those taken from the Mil-Spec fuel Test 12E. Figures 18 and 19 show a control and test sample taken from the soil under the engine.

Here the control shows the absence of a measurable peak in the time frame of 9.6 to 10.2 minutes but the test sample (figure 19) has a very definite peak. The area of this peak indicates a concentration in the soil of very close to 1 ppm. Here again it can be seen that the two orders of magnitude purification step produces a corresponding change in the soil impurities found.

Additional tests were run on soil samples taken at greater distances from the engine centerline. The results using purified fuel and looking at control and test samples from 1.5 to 3.0 meters away from centerline showed only trace amounts of aniline. Whereas, comparison of a control and test pair from a firing using Mil-Spec fuel showed almost an order of magnitude increase in the amount of aniline in the soil (0.001 to 0.004 vs 0.01 to 0.004 ppm) at a distance of 1.5 meters from the centerline.

Two very important points can be drawn from this phase of the analysis. The first is that the aniline does pass through the engine and reaches the soil in high enough concentration to interfere with the organic analysis experiment. The second is that the purification process reduces the aniline contribution in the soil such that it becomes non-interfering with the science objectives.

Ultraviolet light-plume gas interaction studies - Although it is virtually impossible to simulate a true Martian atmosphere in the tests conducted at White Sands the important factors (low pressure, soil, atmospheric composition, etc.) were incorporated in this program. From the very beginning of the planning stages for the Phase II program, it was apparent that one major factor on Mars could not be included in the vacuum chamber tests. The surface of Mars is exposed to a strong flux in the UV from approximately 1900 A upward. This flux could play an important role in either the synthesis or distribution of the plume gas constituents and could not be ignored in a study of the site alteration problem.

A study of the interaction products of a simulated plume gas mixture under ultraviolet light similar to that calculated for the surface of Mars was conducted by the Martin Marietta Research Institute for Advanced Studies (RIAS) division. A report of the details of this work is included in reference 14. In general, this study has shown that in the short time that the original plume gas remains around the lander (up to 3 days) no organic synthesis takes place. That is, either there is an absence of conditions necessary for synthesis to proceed or the amount of synthesis in this short time is not detectable.

Gas diffusion studies - One of the basic experiments in the Viking mission is a sampling of the atmosphere around the lander in order to determine the exact composition of the atmosphere on Mars. This is not only an attempt to measure the major atmospheric constituents but is also an attempt to get information about the less abundant species (to approximately 100 ppm). Thus, gas dissipation from the soil (release

and diffusion) is an important factor in understanding the chemical analysis of the atmosphere. The Viking Molecular Analysis Team suggested a criterion where they would accept a level of contamination above the 10 ppm for up to 2 days after the landing event. This time would be sufficient for diffusion of the plume gases (actual calculated time is 2.7 days, but any minor wind or air movement will decrease this time).

In order to obtain some experimental data on just how long it will take for the plume gases such as ammonia and hydrogen to escape from a soil after engine cutoff a study program was initiated at the Martin Marietta Corporation, Denver Division. In this program, measured amounts of gas were added to a soil sample and the rate of release or gross weight change was determined. The exact experiment procedure and test conditions are described in reference 15. There was no weight loss measured over a 2-day period from soils containing 100-500 ppm of ammonia which indicates the impurities will remain in the soil for an extended time. The only drawback to the experiment was that weight losses of less than 15 ppm were outside the sensitivity limits of the experiment apparatus. This combined with the fact that a 1 ppm weight loss produces an atmospheric contamination of more than 100 ppm means that this source of contamination could negate any attempts to measure atmospheric nitrogen or ammonia at these low concentrations.

Miscellaneous tests - A few special studies that either had direct bearing on the site alteration program or were indirectly related to some of the atmospheric gas impurities were carried out parallel to the major study effort. These included work done by the members of the Molecular Analysis Team on the soils taken from the chamber before and after the tests, by the analysis staff at White Sands, and by other groups at the Martin Marietta Corporation, Denver Division.

In summary, the studies conducted by the Molecular Analysis Team members showed a trace of aniline in the soil from the Mil-Spec fuel tests (small m/e 93 peak in the mass spectrum) and an increase of ammonia between the control and test runs. Attempts to get quantitative measurement for amino acids, hydrazine, aniline or hydrogen cyanide from the soil samples met with failure. The tests, because of their detection limits and the fact that the basic test soil contained a large number of organics at low concentration, did not provide conclusive evidence that some organics were not added to the soil. The data did show that there were no compounds formed in a concentration of greater than 10 ppm.

The chemical staff at White Sands ran tests where soil was heated either directly in the source of a low resolution mass spectrometer or in a column in front of a gas chromatograph/mass spectrometer inlet system. The data served only to verify the results of the Molecular Analysis Team. That is, there were differences between tests run with and without purified fuel but the concentration of the new peaks and their identification were impossible to determine because of the small amounts involved and the relatively high background.

The only analysis specifically for the determination of hydrazine reported that less than 5 ppb of this fuel could be found in the soil. This is in accord with the assumption that the hydrazine fuel is decomposed in a hot catalyst bed to greater than 99.9%. Another large source of hydrazine fuel is the fuel tanks on the aeroshell and on the lander. The tanks on the lander are shut off upon landing and are designed to be a negligible source of hydrazine. The aeroshell will, however, impact the Martian surface in a free fall and the tanks carrying some 4.5 kg of hydrazine could rupture. However, analysis (reference 16) has shown that this source has less than one chance in a thousand of contaminating the landing site.

Conclusion - The interaction of the rocket plume field with the soil does contaminate the site for chemical analysis. The studies carried out during this program allow for a better understanding of how much and what sources are important in this role. The largest and most serious type of contamination from purified hydrazine fuel is the 100-500 ppm of ammonia found in the soil. The gas diffusion studies showed that in the lunar nominal soil the ammonia was not released rapidly from the soil (less than 1 ppm/hr). If the ammonia does come out of the soil at this low rate, it could contaminate the atmosphere around the lander. The presence of ammonia complicates both the experiment preparation for Martian chemical analysis and the interpretation of the returned data. The primary concern is the potential reaction of ammonia with simple organics to form nitrogen-containing organics which would add to the difficulty in interpretation. Further, these new compounds could be of a biological nature and of the type considered primary for a life searching mission.

The aniline found in the soil from Mil-Spec fuel tests would contribute to the difficulty in the interpretation of the data. Its concentration in the sampling site could be even higher if the engine plume passed over that area before landing. Although the single peak of aniline in a chromatogram could be disregarded during the data reduction steps, this compound could mask the presence of other organics in the spectrum. The aniline is associated with the hydrogen cyanide in the plume gases. Both problems can be eliminated by the use of purified fuel. The data show that both the aniline and the hydrogen cyanide levels are below detectable limits when the purified fuel is used.

All other compounds appear to be of little consequence in the site alteration problem studied. Additional work should be carried out to determine how much nitrogen is picked up by the soil around the landing site. More important, the amount and time constant of nitrogen release is a primary question which must be answered before complete interpretation of the atmospheric analysis experiment can be accomplished. The ammonia release is a related but somewhat less important question. Additional sources of contamination such as trapped fuel have been considered and related to the basic plume contamination (reference 16).

Biological Alteration

Lunar nominal soil population - The determination of the microbe populations of the soil from eight sampled barrels indicated that lunar nominal contained sufficient numbers of microorganisms for the ensuing tests for biology site alteration. On aerobic trypticase soy agar (TSA) plates the average number of colony-forming units/gm of soil for triplicate plates ranged from 13,000 to 79,000 for six of the eight samples. The anaerobic thioglycollate plates had a range of 3100 to 9300 for five of the eight samples.

Effect of evacuation and carbon dioxide atmosphere - Simulated evacuation: Half-gram soil samples were used as control and test samples in a simulated test chamber evacuation. The evacuation sequence was simulated using a vacuum desiccator and the backfill sequence was accomplished with carbon dioxide and nitrogen gases. The desiccator was pressurized to 5.17 Torr with carbon dioxide over a 10-minute period and after 15 minutes leaked to 7.2 Torr. The desiccator was pressurized to ambient pressure with nitrogen. The data from the simulated evacuation and backfill with carbon dioxide showed that in all cases the vacuum and carbon dioxide environments did not alter the biology sufficiently to violate the criterion. For TSA the t-test indicated acceptance of equal means for control and test samples for barrels 5, 10, and 13. For thioglycollate the means of control and test samples of barrels 5, 6, 13, 21, 99 and 123 were accepted as equal.

Trial run at WSTF 302 chamber - Soil samples were taken from the biology tray inside the vacuum chamber at WSTF before and after the Trial Run 12A. The soil from barrel 5 had been transferred to the biology tray and the bed had been prepared and covered the day before. Just prior to closing the chamber doors the bed was uncovered and surface samples were taken at 1.5, 2.4 and 3.4 m from the centerline of the engine at the left, center and right sides of the biology tray. After the Trial Run 12A, samples were taken as above.

The results of the trial run are presented in figure 20 and in every case for TSA the means of control and test samples were accepted as equal. For thioglycollate 2 of the 3 sets passed the t-test for equal means and the third met the biology criterion of less than 50% population reduction.

Effect of engine firing tests - Pure Fuel: Soil samples were taken from the biology tray before and after the engine firing of Test 12A. In addition, one control sample was taken from the main bed at 1.5 m from the engine centerline prior to firing and test samples were obtained from the main bed after firing at the same point as the control and 0.15 m from the centerline of the engine. Surface samples were obtained before and after firing in the same manner as described for the trial run. Core samples were taken before and after firing at the same points as the surface samples. The test results for Pure Fuel Test 12A are shown in Figure 21.

Surface samples were taken for the four Viking biology experiments (Gas Exchange, Labeled Release, Light Scattering and Pyrolytic Release) from the left sampling areas at 1.5 and 3.4 m from the engine centerline (see figure 21) before and after engine firing.

Only three of the 16 test sample means were rejected by the t-test as being less than the corresponding control means. Those three were for the 0.15 m centerline TSA and 2.4 m core sample in both TSA and thioglycollate; however, they did meet the biology criterion. Each sample mean for the biology tray in this experiment was calculated from 7-9 different sample counts from 3 different surface or core samples at a given distance from the engine centerline.

For Test 12C surface soil samples were taken before and after engine firing. These control and test samples were taken at 0.15, 1.7 and 2.9 m from the engine centerline as shown by the crossmarked areas in figure 22. The soil samples for the four Viking biology experiments were obtained from the top few millimeters of soil between the crossmarked areas at 1.7 m from the engine centerline. The test results for the Pure Fuel Test 12C are shown in figure 22.

Only one test mean (1.7 m R TSA) was rejected as being less than the corresponding control mean for the test, but it did meet the biology criterion. Each sample mean was calculated from 6 different sample counts from two different samples at a given distance from the engine centerline with the exception of the 2.9 m thiogly-collate control samples which had unreasonably high counts for the right side of the tray.

Mil-Spec Fuel: Surface soil samples were taken before and after engine firing for Mil-Spec Fuel Test 12D. These control and test samples were taken at 0, 1.5 and 2.7 m from the engine centerline as shown by the crossmarked areas in figure 23, which also shows the results of this experiment. The soil samples for the four Viking biology experiments were obtained from the top few millimeters of soil between the crossmarked areas at 1.5 m from the engine centerline.

Two of the six test means (centerline TSA and 2.7 m TSA) were rejected as being less than the corresponding control mean for the tests, but they met the biology criterion. Each sample mean was calculated from 5 or 6 sample counts from two different samples at a given distance from the engine centerline.

<u>Viking experimenters' tests</u> - Initial work done by the Viking biology experimenters was centered around the possible effects of hydrogen cyanide on different soil samples. Although it was very clear from the beginning that this approach would be inadequate in leading to a true understanding of the effects of hydrogen cyanide on soil microbiology, the tests would lead to a better and more general understanding of this problem.

Eight different soil samples were collected by researchers at the NASA/Ames Research Center and treated with 500 ppm of hydrogen cyanide in carbon dioxide for 1280 minutes. Eight control samples were created at the same time by exposing equal amounts of each soil to 100% carbon dioxide atmosphere for 1280 minutes. Each biology experimenter received sixteen samples (8 control and 8 test) which were run using their laboratory experimental equipment. Since the four Viking biology experiments (Gas Exchange, Labeled Release, Light Scattering and Pyrolytic Release) are so different, it was felt that this approach would be the best proof that the hydrogen cyanide did or did not affect the results.

The results of these tests showed that there was little difference between the control and test samples. Those differences which were detected were only a factor of 1.5 to 2 lower in growth than the corresponding controls. However, all experimenters reported that these differences were within the normal 'plate counting' analysis by the NASA/Ames group. The results of these tests were in general agreement with the results presented by the four experimenters but the variation was slightly larger (factor of 2 or 3).

The same general approach was used in the analysis of the biology soil samples taken during the site alteration tests. The samples were taken from the biology soil tray from the same position and at the same time as the samples reported in the earlier part of this section. These samples were mailed to the experimenters and each control and test pair were run in the laboratory version of each of the four experiments. Results of these tests have shown that there was little or no difference between each pair.

Conclusion - The Viking Mission Definition (reference 17) shows the type of experiments that will be carried out on the surface of Mars. The mission definition also brings out some of the requirements that the experiments impose on the terminal descent landing phase, but because of its brief nature, it does not accomplish this fully. A more detailed set of requirements has been generated and these can be found in reference 4. The site alteration criteria defines for both the molecular analysis and biology investigations a level of acceptability for the degree of site alteration.

In all of the biology related engine firings with Mil-Spec or purified fuels at WSTF the biology criterion was met. This was concluded from the fact that the one-sided t-test at the 95%-confidence level did not show that the microorganism population in the soil was reduced more than 50%.

The most important fact that came from all of the biological test data is that the combined results of soil erosion, thermal exposure and plume gases did little to change the basic microorganism population in the soil. Although these results cannot be directly related to biological response of true Martian soil, it does indicate that there is a reasonable probability that microorganisms on Mars could survive the landing event.

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NOTE:

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References 8 and 17 available from NASA Langley Research Center, Hampton, VA.

TABLE I. SUMMARY OF WSTF PHASE II TEST CONDITIONS

				1		Ţ	ROCKET ENGINE									
		Test	Toront	Test Chamb Press Initial	nie		ber rature	Nozzle		Burn	Chamber	Max. Chamber tempern- ture.	Descent	soil s		
Test no.	Test objectives	date	Target surface	mb,	October,	K K	Curoff,	Configura tion	N N	time, sec	N/m ²	og.	m/sec	Initial m	COLOTT	Hydrazine
1	Engine Checkout - Flight Weight	3/1/71		Amb.	Amb.	283.81	284.57	Baseline	533.8	10.25	379 x 10 ³		0	3.048	3.048	Pure
2	Pure Hydrazine - Exhaust Gas	3/5/71	Quartz Plate	3,30	11.80	290.11	290.61	"	640.5	15.55	456		0	3.048	3.048	Pure
3		3/9/71	Quartz Plate	9.93	21.20	287.60	288.10	"	636.1	15.40	452		0	3.048	3.048	Pure
10A	Test Chamber Chemical Background	3/10/71	•	4.74	4.74											
9	Mil Spec Hydrazine - Exhaust Gas	3/16/71	Quartz Plate	4.47	14.90	289.61	290.86	Baseline	630.1	15.35	451		0	3.048	3.048	Mil Spec
10	, , ,	3/17/71	Quartz Plate	10,00	22,10	288.61	289.86	"	649.4	15.65	461		0	3.048	3.048	Mil Spec
1A	, ,	3/26/71		Amb.	Amb.	291.61	292.11	"	622.7	10.44	345	1103.60	0	3.048	3.048	Mil Spec
4	Checkout of Rail Assembly	3/31/71		Amb.	Amb.								1.453	11,131	4.639	- -
5	•	3/31/71		Amb.	Amb.								1.435	11.131	1.359	
11		4/2/71	Flat Plate	11.50	19.90	292,35	293.10	Baseline	653.9	10.93	361	1114.71	1,435	12,161	1.652	Mil Spec
11A	Pressure Test	4/6/71	Flat Plate	9.25	20.10	284.58	285.34	24	590.0	10.20	421		1.444	12.344	1.981	Mil Spec
11C		4/7/71	Flat Plate	11.30	19.00	291.11	291.61	7	596.0	7.50	432		1.456	12.293	5.486	Mil Spec
		4/16/71	Stainless Steel Plate	1.94	15.90	289.61		Baseline 24	587.1	16.50	325 472	1074.15	0	3.048	3,048	Pure
	Pure Hydrazine - Exhaust Gas	4/19/71	Stainless Steel Plate	H.94	.'1.62	282.80			602.8	16.21		1078.60	0	1.524	1.524	Pure
3A	·	4/20/71	Stainless Steel Plate	2.77	14.30	289.11		7	671.7	16,34	475	1081.38	0	1.524	1.524	Pure
		4/26/71		Amb.	Amb.	293.07		2. (mod 1)	569.3	10.16	537	1059.71	0	3,048	3.048	Pure
1	į		Plat Plate	11.42	18.70	299.76		24 (mod 1)	508.4	10.38	481	1051.93	1,475	12.374	.640	Pore
ŀ			Flat Plate	11.10	18.80	296.32		24 (mod 2)	508.4	10.39	480	1044.16	1.639	12.38/	.804	Pure
ŀ	Effects		Lunar	11.58	19.40	299.51	307.77	24 (mod 2)	647.2	9.33	630	1068.60	1.5+0	12.274	.691	Pure
11G	i	5/21/71	Flat Plate	4.74	18.70	292.11	300.00	Multi-7-D	715.2	10,43	528	1120.82	1,427	12,292	.688	Pure
1111	:	5/25/71	Plat Plate	10.10	18.90	291.61	299.51	Annular	725.5	10.05	44 /	950.28	1.600	12.301	.707	Pure
111			Flat Place	100	19.80	293.50	301.47	Fluted	743.7	10.38	541	1136.37	1.530	12.28t	.722	Pore
12C	Soil Cratering, Chemistry Effects	6/5//1	Luner	17,66	19.70	310.41	317.28	24 (mod 2)	738.8	9.58	,og	1117.48	1.578	12.274	.691	Pure
Ì	Soil Cratering, Chemistry Effects	6/15/71	Lunar	10.00	19.00	31.1.79		24 (mod 2)	685.0	10.05	664	1114.71	1.000	12.274	.691	MII Spec
	Soil Cratering, Chemistry Effects	-	Lunar	10.80	18.70	311.62	315.67	18	U65.0	9.99	463	1106.37	1.609	12.252	. 658	Mil Spec
12F	Soil Cratering	6/28/71	Dune Sand	11.90	14.60	312.06	316.12	18	067.2	9.87	465	1108.04	1.439	12.252	.658	Mil Spec
12G	Soil Cratering	7/4/71	Dune Sand	11.55	19.80	104.88	108.01	Fluted (mod 1)	681.4	9.77	503	1154.15	1,658	11.205	.633	Mil Spec
12#	Soil Cratering, Ammonia	7/13/71	Dunc Sand	11.32	18.80	30 57	308.97	Flured (mod 1)	605.4	8,40	487	1139,70	1.509	11.205	1.942	Mil Spec
iIJ	Pressure Test	7/20/71	Flot Plate	9, .0	17.90	296.56	301.73	Fluted (mod 1)	666.8	14.24	497	1148.59	1.600	12.225	.722	Mil Spec
11K	Pressure Test	7/22/71	Flat Plate	10.32	19.30	295.56	301.23	18	685.0	10.14	451	1075,82	1.557	12.283	. 7/12	Pure
29	Characteristics of Engine Plume	7/28/71	Flat Pl. te	9.47	19.10	297.06	302.67	18	673.9	14.26	469	1098.04	1.557	12.283	. 162	Pure

TABLE II. CERTIFIED ANALYTICAL REPORT* ON WSTF SOIL SAMPLES
(From Soil Control Lab, Watsonville, California)

(11 on boil control hab, watsonville, California)								
	SOIL IDENTIFICATION NUMBER**							
Chemical Components	WSTF-5	WSTF-8	WSTF-123					
Moisture, percent @ 110°C	0.78%	0,71%	0.73%					
pH Value, units (as paste in water)	9, 35	8. 95	9, 00					
Redox Potential, volts (as paste)	+0.009	+0.009 +0.002						
Soluble Carbonate Carbon (c)	0 . õ	0.4	0.4					
Soluble Bicarbonate Carbon	8.1	7.0	6, 9					
Total Inorganic Carbon	72. 0	36, 0	84.0					
Organic Carbon (by difference)	240.0	288,0	240.0					
TOTAL CARBON'	312.0	324.0	324.0					
Ammonia Nitrogen (N)	0.0	0.0	0.0					
Nitrate Nitrogen	4. 0	5. 0	5 . 0					
Nitrite Nitrogen	0.0	0.0	0.0					
Organic Nitrogen (by difference)	19.0	13.0	8.0					
TOTAL NITROGEN (Kjeldahl)	23.0	18,0	13, 0					
Soluble Sulfide Sulfur (S)	0.0	0.0	0.0					

^{*}Quantitative chemical analysis expressed as parts per million by weight where not otherwise indicated.

^{**}Hazen soil barrels as numbered above.

TABLE III. AMMONIA MEASUREMENTS IN SOIL

Distance from Engine Centerline, m	Consentration of NH3, 10-5 gm/gm of soil for tests								
	12A	12C	12D	12F	12 F	12G	12H		
0	240*	170*	-	498*	-	-			
1.5	12	46	11	-	343*	34	63		
	2	270*	50	728*	290*	37	35		
	5	-	-	-	-	61	86		
3. 0	23	-	56	<u>-</u>	_				
	10	22	56	-	-	98**	116**		
	4	_	_	_					
4 . 6	17	1	51			45	67		
	13	25	43	-	-	300*	235*		
	7	-	-	-	-	-	-		
ö . 1	-	-	43	-	-	-			
	-	36	32	-	-	-			
Control	1	-	-	-		-	-		
	1	5	1	2	-	-	-		
	-	-	-	-	-	-	_		

^{*}Soil exposed for 10 minutes

TABLE IV. CYANIDE MEASUREMENTS IN SOIL

Distance from Engine Centerline, m	Concentration of HCN, 10 ⁻⁸ gm/gm of soil for tests								
	12A	12C	12D	12E	12F	12G			
0	112.0	6.4*	-	8.4*		_			
1.5	0.1	10.0		~		10.1			
	0.3	1.2	1.5	3.8*	5. 4	14.1			
	0, 3	-	-			15.3			
3.0	0.4	_	1.6			-			
	0.4	, 4.1	2.0	-	-	5, 6			
	0.2	_	_	_					
4 . ú	0. 6	-	3.1	_	_	G. 6*			
	0, 2	2.0	1.7	_	_				
	0.4	-	1	_	-				
6. 1	-	-	1.9	-	_	_			
		1.2	2.0	-					
Control	0.6	-		-					
	0.1	1.0	0.7	0.5	-	_			
	0,4	-	1	_		-			

^{*}Open for more than 10 minutes after firing

^{**}Soil in direct plume flow field

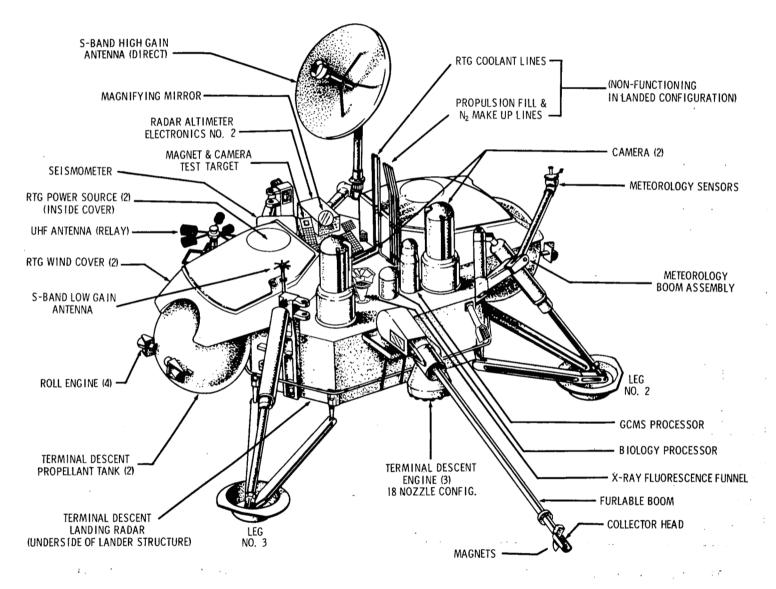


Figure 1. - Viking Lander Configuration.

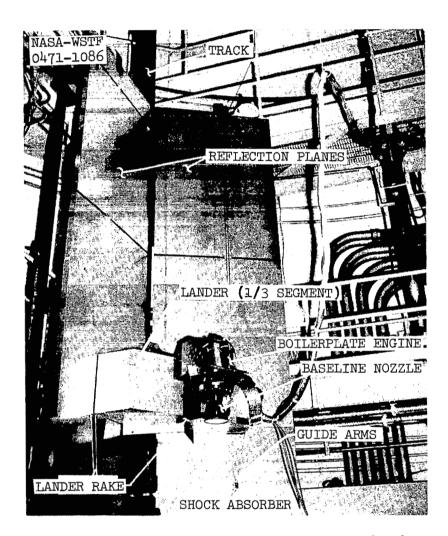


Figure 2. - Phase II test apparatus in WSTF chamber.

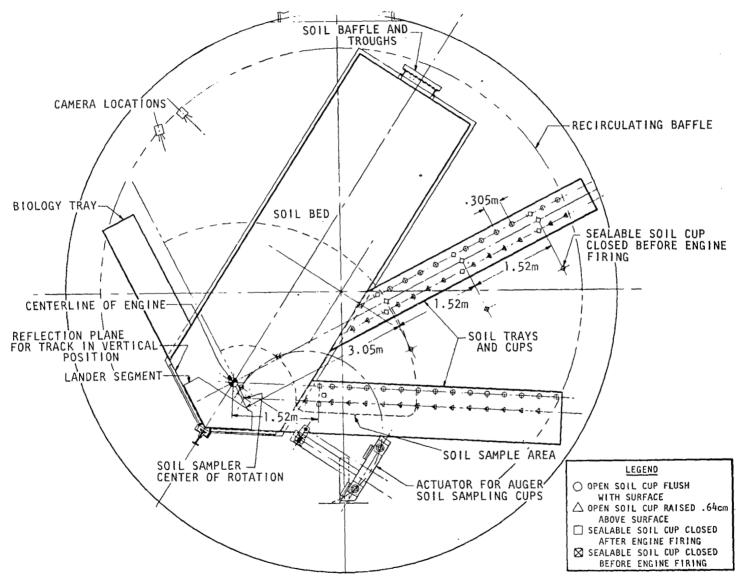


Figure 3. - Location of soil bed, trays and cups in WSTF 302 test chamber.

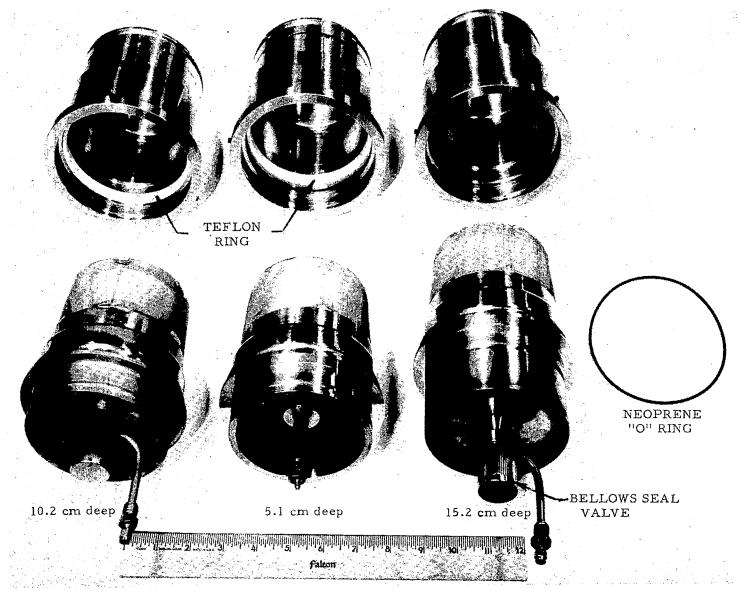
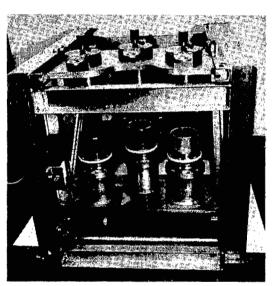


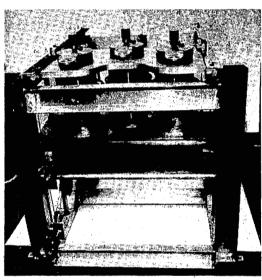
Figure 4. - Sealable soil cups.



(a) Initial position of cup carriage and cap carriage



(b) Position of carriages at end of 1st command to seal



(c) Final position of carriages with soil cups sealed

Figure 5. - Actuator assembly used to seal 1 to 3 soil cups.

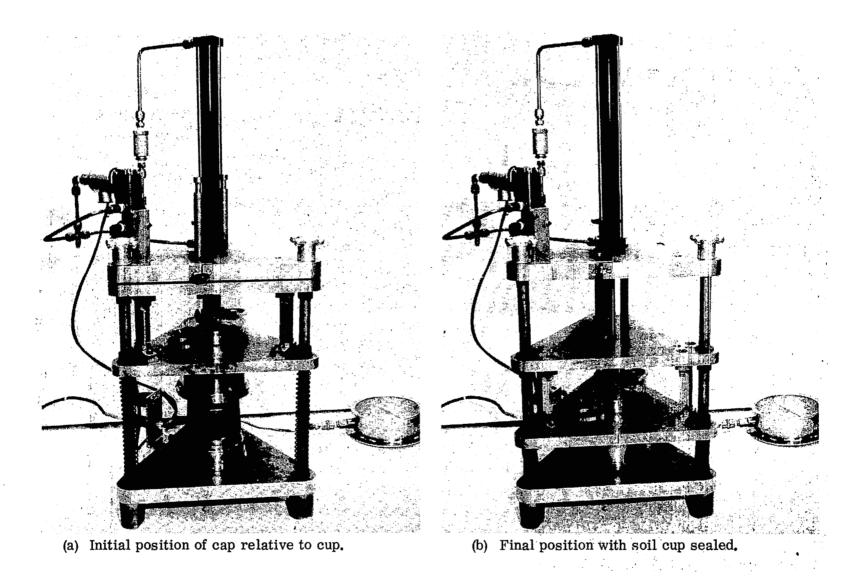
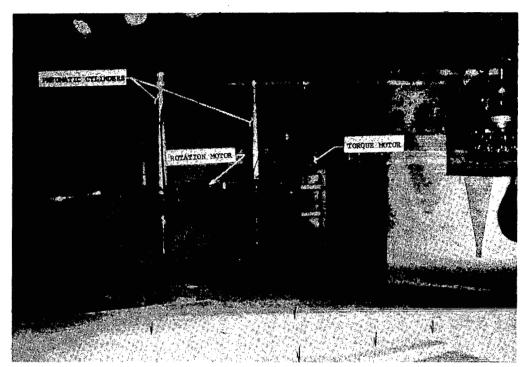
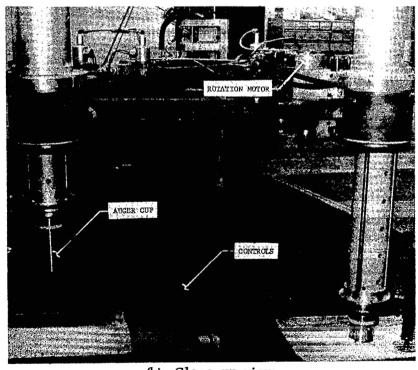


Figure 6. - Actuator assembly used to seal single soil cups.



(a) Installation in 302 chamber.



(b) Close-up view.

Figure 7. - Auger assembly.

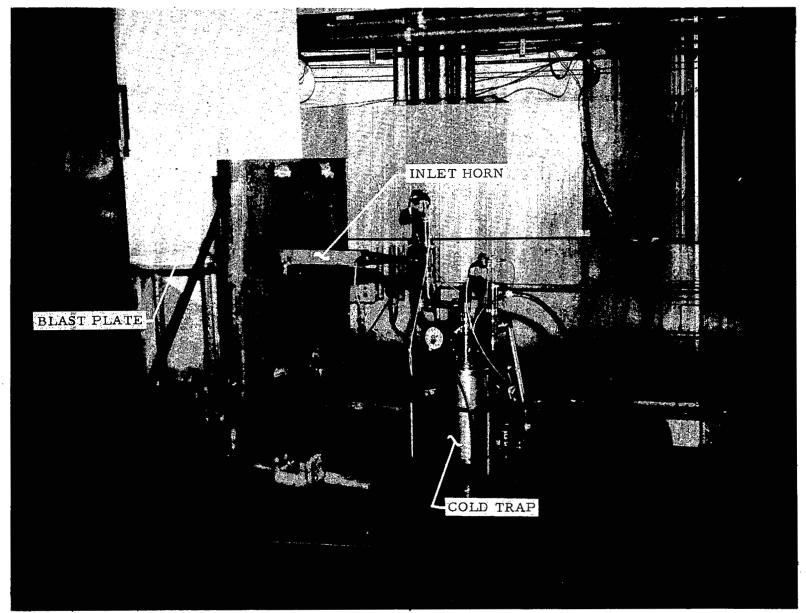
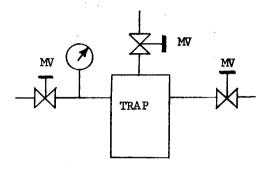


Figure 8. - Exhaust gas sampling equipment.



LABORATORY SAMPLER CONFIGURATION

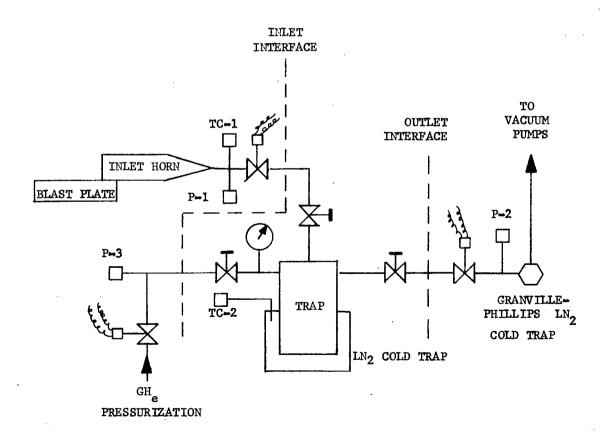


Figure 9. - Exhaust gas sampling schematic.

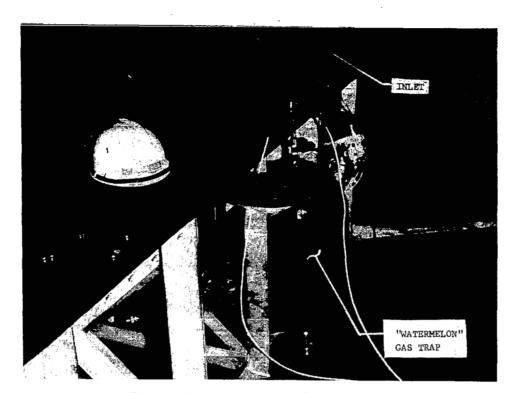


Figure 10, - "Watermelon" gas trap.

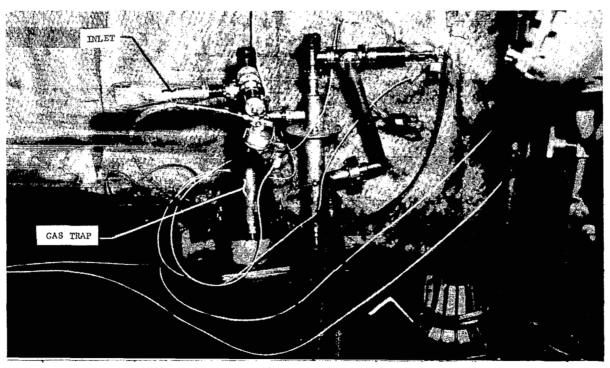


Figure 11. - Exhaust gas sampling equipment.

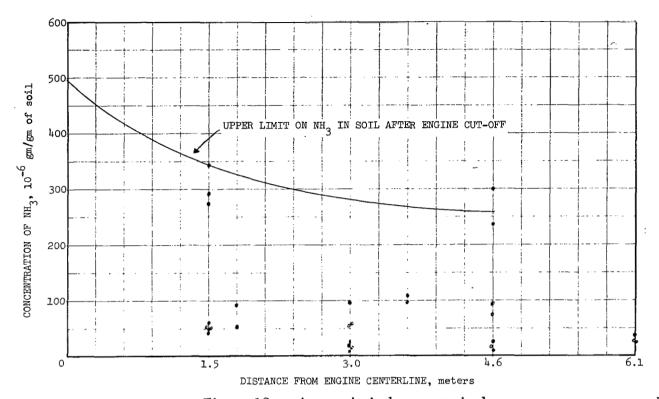


Figure 12. - Ammonia in lunar nominal.

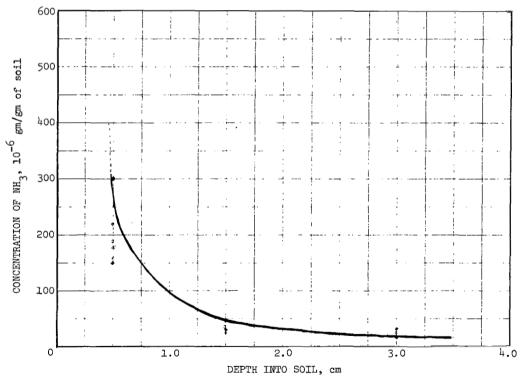


Figure 13. - Ammonia concentration variation with depth of soil.

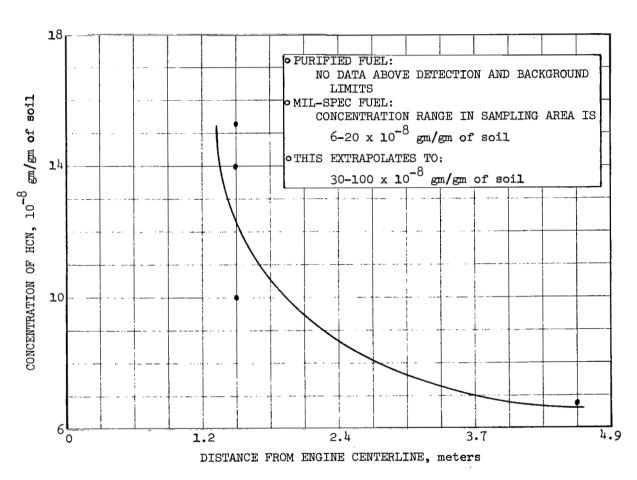


Figure 14. - Hydrogen cyanide in lunar nominal.

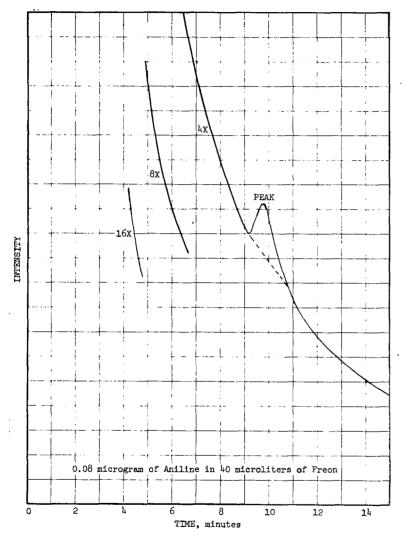


Figure 15. - Gas Chromatogram of aniline calibration standard.

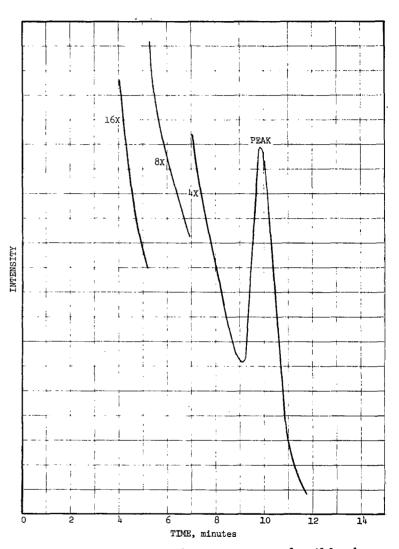


Figure 16. - Gas Chromatogram of soil bank and analine.

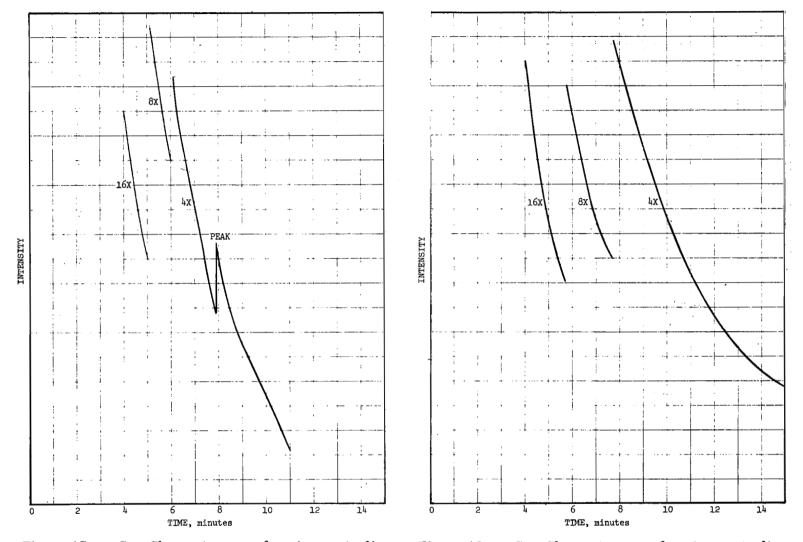


Figure 17. - Gas Chromatogram of engine centerline soil test sample (purified fuel).

Figure 18. - Gas Chromatogram of engine centerline soil sample sample.

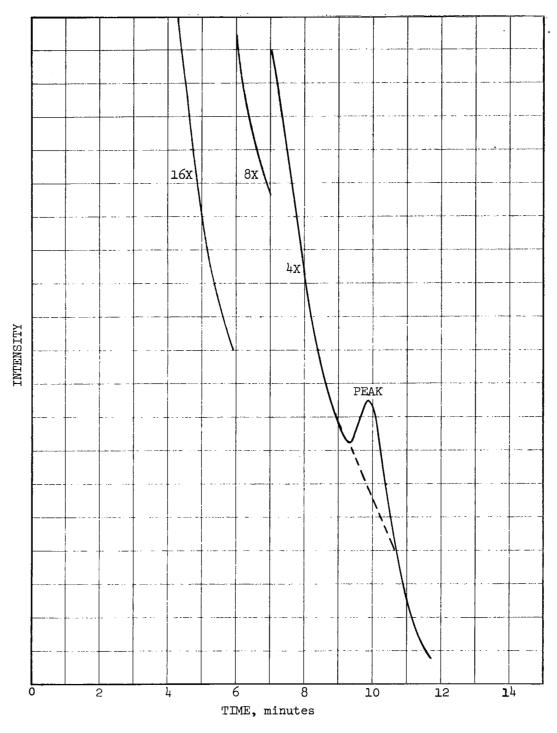


Figure 19. - Gas Chromatogram of engine centerline soil test sample (purified fuel).

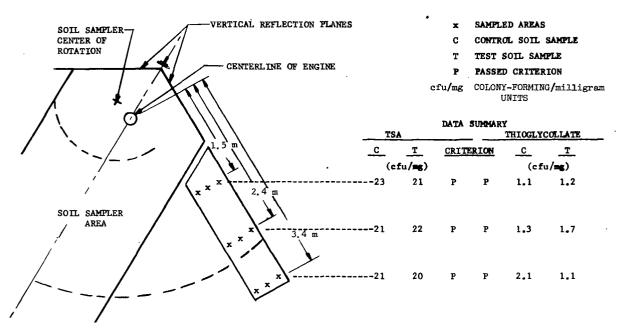


Figure 20. - Trial run 12A, biology.

SAMPLED AREAS
CONTROL SOIL SAMPLE

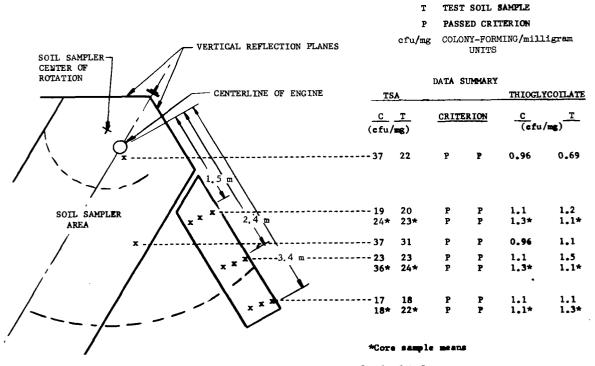


Figure 21. - Test 12A, pure fuel, biology.

- x SAMPLED AREAS
- C CONTROL SOIL SAMPLE
- T TEST SOIL SAMPLE
- P PASSED CRITERION

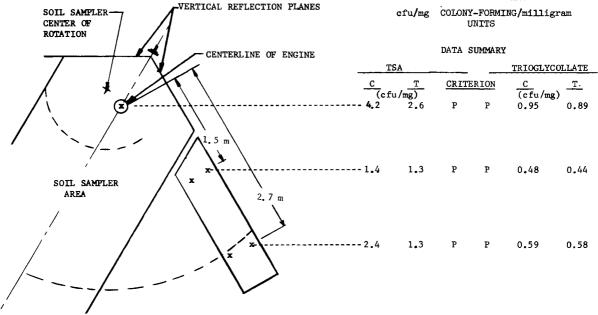


Figure 22. - Test 12C, pure fuel, biology.

- x SAMPLED AREAS
- C CONTROL SOIL SAMPLE
- T TEST SOIL SAMPLE
- P PASSED CRITERION

cfu/mg COLONY-FORMING/milligram -VERTICAL REFLECTION PLANES SOIL SAMPLER CENTER OF units ROTATION CENTERLINE OF ENGINE DATA SUMMARY TRIOGLYCOLLATE CRITERION (cfu/mg) 19 0.88 0.80 1.7 m 1.4 2.9 m -----22 26 0.84 1.1

Figure 23. - Test 12D, Mil-spec fuel, biology.

1